Supplementary Material (ESI) for Chemical Communications

Supporting Information

First iron-catalyzed synthesis of oximes from styrenes

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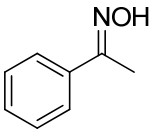
General

Unless otherwise indicated, all chemicals were obtained from commercial suppliers, and were used without further purification. All reactions were performed under an atmosphere of argon. Toluene and DME were purchased from Acros and distilled from sodium. 2-Propanol was used without further purification (purchased from Fluka, dried over molecular sieves). Flash chromatography was performed with FLUKA Silica gel 60 (70-230 mesh) in common glass columns. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AV300/AV400 spectrometer. Chemical shifts (δ) are given in ppm and refer to TMS or the residual undeuterated solvent as the internal standard. Gas chromatography was performed on a Hewlett–Packard HP 6890 chromatograph with a 30 m HP5 column. EI mass spectra were recorded on an AMD 402 spectrometer (70 eV, AMD Intectra GmbH). IR spectra were recorded on an FTIR Nicolet 6700 (Thermo ELECTRON CORPORATION). Melting points were measured with a Stuart melting point apparatus (SMP3) and are not corrected.

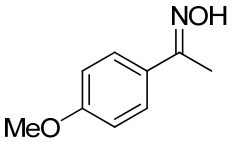
General procedure for the preparation of oximes

An autoclave was charged under argon atmosphere with iron phthalocyanine (Fe(Pc), 6 mg, 0.01 mmol), $NaBH_4$ (76 mg, 2 mmol), *t*-butyl nitrite (0.3 mL, 2.5 mmol), the corresponding olefin (3 mmol) and absolute EtOH (18 mL). Then, the autoclave was filled with 10 bar of H_2, and the mixture was stirred for 3 h at room temperature. The reaction mixture was neutralized with 1N HCl and extracted with ether (3 x 25 mL). The combined organic extracts were dried over Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel with gradients EtOAc/hexanes as eluent to yield the oxime products.



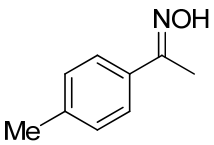
Acetophenone oxime (2).[1-2,4]

White solid; yield 79% (320 mg), mp 58-60 °C. IR ν_{max} (KBr)/cm^{-1} 3235, 3084, 2924, 1496, 1444, 1370, 1300, 1079, 1005, 924, 847, 773, 756, 688, 651; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.86 (br s, 1H), 7.69-7.58 (m, 2H), 7.43-7.36 (m, 3H), 2.32 (s, 3H), ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.1, 136.6, 129.3, 128.6, 126.1, 12.3; MS (EI) *m/z* (% rel. intensity) 135 (M^+, 86), 118 (14), 106 (22), 103 (26), 94 (32), 77 (100), 66 (16), 51 (33); HRMS (EI): calcd C_8H_9NO: 135.0679; found: 135.0683.



1-(4-Methoxyphenyl)ethanone oxime (4a).[1,3-4]

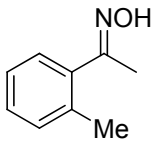
White solid; yield 65% (320 mg), mp 85-87 °C. IR ν_{max} (KBr)/cm^{-1} 3208, 3077, 3007, 2966, 2936, 2839, 1609, 1515, 1297, 1257, 1174, 1023, 920, 829, 748; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.26 (br s, 1H), 7.60 (d, J = 8.9 Hz, 2H), 6.93 (d, J = 8.9 Hz, 2H), 3.85 (s, 3H), 2.31 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.5, 155.6, 129.1, 127.4, 113.9, 55.4, 12.2; MS (EI) *m/z* (% rel. intensity) 165 (M^+, 100), 148 (44), 134 (41), 134 (41), 108 (38), 92 (20), 77 (38), 63 (15); HRMS (EI): calcd $C_9H_{11}NO_2$: 165.0784; found: 165.0789.



1-(4-Methylphenyl)ethanone oxime (4b).[1,4]

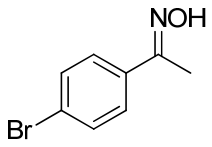
White solid; yield 69% (310 mg), mp 84-86 °C. IR ν_{max} (KBr)/cm^{-1} 3290, 3204, 3127, 3057, 3031, 2918, 1610, 1514, 1436, 1364, 1313, 1303, 1187, 1117, 1009, 921, 812, 748, 712; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.23 (br s, 1H), 7.54 (d, J = 8.3 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 2.38 (s, 3H), 2.30 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.0, 139.3, 133.7, 129.3, 126.0, 21.3, 12.3;

MS (EI) *m/z* (% rel. intensity) 149 (M^+, 100), 132 (17), 118 (29), 107 (22), 91 (87), 89 (19), 79 (12), 65 (28), 39 (12); HRMS (EI): calcd $C_9H_{11}NO$: 149.0835; found: 149.0838.



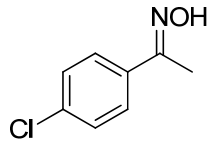
1-(2-Methylphenyl)ethanone oxime (4c).[1,5]

White solid; yield 56% (250 mg), mp 62-64 °C. IR ν_{max} (KBr)/cm^{-1} 3218, 3056, 3021, 2924, 1492, 1433, 1364, 1310, 1273, 1008, 921, 753, 727; ^1H NMR ($CDCl_3$, 300 MHz) δ 9.14 (br s, 1H), 7.46-7.14 (m, 4H), 2.41 (s, 3H), 2.27 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 158.1, 137.4, 135.7, 130.7, 128.6, 128.2, 125.9, 20.1, 15.9; MS (EI) *m/z* (% rel. intensity) 149 (M^+, 50), 134 (98), 132 (59), 117 (100), 115 (47), 91 (48), 89 (25), 77 (15), 65 (36), 63 (19), 51 (13), 39 (14); HRMS (EI): calcd $C_9H_{11}NO$: 149.0835; found: 149.0835.



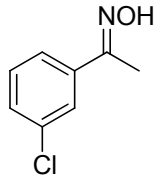
1-(4-Bromophenyl)ethanone oxime (4d).[4]

White solid; yield 82% (530 mg), mp 128-130 °C. IR ν_{max} (KBr)/cm^{-1} 3220, 3084, 2922, 1590, 1487, 1395, 1365, 1310, 1007, 928, 822, 776, 750; ^1H NMR ($CDCl_3$, 300 MHz) δ 8.79 (br s, 1H), 7.56-7.48 (m, 4H), 2.29 (s, 3H), ^{13}C NMR ($CDCl_3$, 75 MHz) δ 155.3, 135.4, 131.7, 127.6, 123.6, 12.1; MS (EI) *m/z* (% rel. intensity) 213 (M^+, 100), 196 (22), 172 (22), 155 (62), 106 (28), 102 (39), 75 (46), 65 (14), 58 (14), 50 (31); HRMS (EI): calcd C_8H_8BrNO: 212.9784; found: 212.9789.



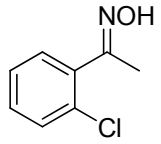
1-(4-Chlorophenyl)ethanone oxime (4e).[4]

White solid; yield 73% (370 mg), mp 97-99 °C. IR ν_{max} (KBr)/cm^{-1} 3291, 3226, 3203, 3062, 2926, 1595, 1493, 1396, 1370, 1311, 1097, 1006, 926, 822; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.99 (br s, 1H), 7.58 (td, *J* = 8.7, 2.3 Hz, 2H), 7.38 (d, *J* = 8.7, 2.3 Hz, 2H), 2.30 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 155.2, 135.3, 134.9, 128.8, 127.4, 12.2; MS (EI) *m/z* (% rel. intensity) 169 (M^+, 100), 153 (34), 138 (65), 128 (28), 111 (75), 102 (26), 75 (50), 50 (22); HRMS (EI): calcd C_8H_8ClNO: 169.0289; found: 169.0289.



1-(3-Chlorophenyl)ethanone oxime (4f).[6]

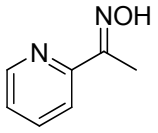
White solid; yield 86% (440 mg), mp 88-90 °C. IR ν_{max} (KBr)/cm^{-1} 3284, 3238, 3073, 2921, 1689, 1562, 1422, 1368, 1291, 1083, 1005, 937, 882, 787, 711, 681; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.89 (br s, 1H), 7.54 (ap t, *J* = 1.8 Hz, 1H), 7.43 (dt, *J* = 7.1, 1.8 Hz, 1H), 7.31-7.21 (m, 2H), 2.21 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 155.1, 138.3, 134.6, 129.8, 129.3, 126.3, 124.2, 12.2; MS (EI) *m/z* (% rel. intensity) 169 (M^+, 100), 152 (79), 138 (26), 128 (48), 111 (93), 102 (30), 75 (62), 50 (25); HRMS (ESI-TOF)(M+H)+: calcd C_8H_9ClNO: 170.0367; found: 170.0365.



1-(2-Chlorophenyl)ethanone oxime (4g).[5]

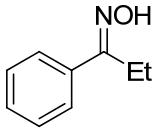
White solid; yield 59% (300 mg), mp 102-104 °C. IR ν_{max} (KBr)/cm^{-1} 3289, 3200, 3142, 3089, 3072, 3059, 2924, 1569, 1431, 1367, 1313, 1018, 928, 743, 722; ^{1}H NMR ($CDCl_3$, 300 MHz) δ

9.08 (br s, 1H), 7.39-7.16 (m, 4H), 2.19 (s, 3H), ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.9, 136.7, 132.6, 130.0, 126.9, 15.8; MS (EI) *m/z* (% rel. intensity) 169 (M^+, 76), 152 (26), 134 (100), 111 (58), 102 (29), 90 (16), 75 (50), 50 (20); HRMS (EI): calcd C_8H_8ClNO: 169.0289; found: 169.0288.



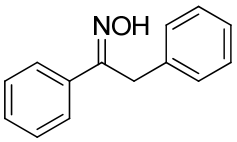
2-Acetylpyridyl oxime (4h).[1]

White solid; yield 59% (300 mg), mp 119-121 °C. IR ν_{max} (KBr)/cm^{-1} 3274, 3156, 3118, 3063, 3045, 2846, 2809, 1591, 1565, 1491, 1440, 1361, 1324, 1284, 1159, 1115, 1002, 935, 833, 786, 778, 751, 673; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.13 (br s, 1H), 8.57 (ddd, 1H, *J* = 4.9, 1.8, 0.9 Hz), 7.77 (dt, 1H, *J* = 8.0, 1.0 Hz), 7.62 (ap td, 1H, *J* = 7.8, 1.8 Hz), 7.21 (ddd, 1H, *J* = 7.4, 4.9, 1.2 Hz), 2.34 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 157.0, 154.3, 149.0, 136.5, 123.8, 120.6, 10.8; MS (EI) *m/z* (% rel. intensity) 136 (M^+, 100), 119 (28), 104 (31), 92 (14), 78 (89), 51 (31); HRMS (EI): calcd $C_7H_8N_2O$: 136.0631; found: 136.0632.



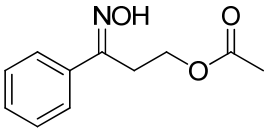
Propiophenone oxime (4i).[1]

White solid; yield 40% (180 mg), mp 56-58 °C. IR ν_{max} (KBr)/cm^{-1} 3275, 3196, 2973, 2940, 2880, 1465, 1455, 1336, 1292, 1101, 1074, 1040, 970, 912, 764, 692; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.48 (br s, 1H), 7.69-7.59 (m, 2H), 7.46-7.37 (m, 3H), 2.86 (q, *J* = 7.37 Hz, 2H), 1.21 (t, *J* = 7.37 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.8, 135.6, 129.3, 128.6, 126.3, 19.8, 11.0; MS (EI) *m/z* (% rel. intensity) 149 (M^+, 70), 148 (73), 132 (67), 117 (21), 115 (16), 104 (73), 94 (15), 91 (31), 77 (100), 51 (31); HRMS (EI): calcd $C_9H_{11}NO$: 149.0835; found: 149.0834.



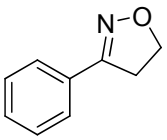
1,2-Diphenylethanone oxime (4j).[1,7]

White solid; yield 44% (280 mg), mp 93-95 °C. IR ν_{max} (KBr)/cm^{-1} 3237, 3084, 3059, 3028, 2912, 1602, 1494, 1452, 1343, 1314, 1062, 1028, 964, 951, 929, 920, 755, 721, 689; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.86 (br s, 1H), 7.59-7.50 (m, 2H), 7.31-7.06 (m, 8H), 4.15 (s, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 157.6, 136.5, 135.6, 129.4, 128.7, 128.6, 128.56, 126.6, 126.4, 32.2; MS (EI) *m/z* (% rel. intensity) 211 (M^+, 40), 193 (99), 165 (21), 120 (10), 103 (19), 91 (100), 77 (45), 65 (18); HRMS (EI): calcd $C_{14}H_{13}NO$: 211.0992; found: 211.0987.



3-(Hydroxyimino)-3-phenylpropyl acetate (4k).

White solid; yield 32% (200 mg), mp 75-77 °C. IR ν_{max} (KBr)/cm^{-1} 3332, 3057, 2961, 1738, 1715, 1445, 1365, 1232, 1033, 970, 937, 915, 759, 693; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.82 (br s, 1H), 7.73-7.66 (m, 2H), 7.48-7.41 (m, 3H), 4.42 (t, J = 7.03 Hz, 2H), 3.24 (t, J = 7.03 Hz, 2H), 2.02 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 171.3, 156.1, 135.5, 129.5, 128.7, 126.4, 60.9, 26.5, 20.9; MS (EI) *m/z* (% rel. intensity) 207 (M^+, 1), 164 (3), 147 (100), 130 (24), 117 (82), 104 (27), 103 (30), 91 (26), 77 (62), 51 (21), 43 (91); HRMS (EI): calcd $C_{11}H_{13}NO_3$: 207.0890; found: 207.0892.



3-Phenyl-2-isoxazoline (5).[8]

White solid; yield 14% (60 mg), mp 68-70 °C. IR ν_{max} (KBr)/cm^{-1} 3194, 3059, 2966, 2894, 1496, 1473, 1448, 1433, 1355, 1185, 926, 881, 850, 758, 694; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.66-7.58

(m, 2H), 7.37-7.30 (m, 3H), 4.42 (t, J = 10.1 Hz, 2H), 3.27 (t, J = 10.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.9, 130.1, 129.5, 128.8, 126.8, 69.2, 35.3; MS (EI) *m/z* (% rel. intensity) 147 (M^+, 100), 117 (53), 91 (24), 77 (43), 63 (10), 51 (21); HRMS (EI): calcd C_9H_9NO: 147.0679; found: 147.0683.

References

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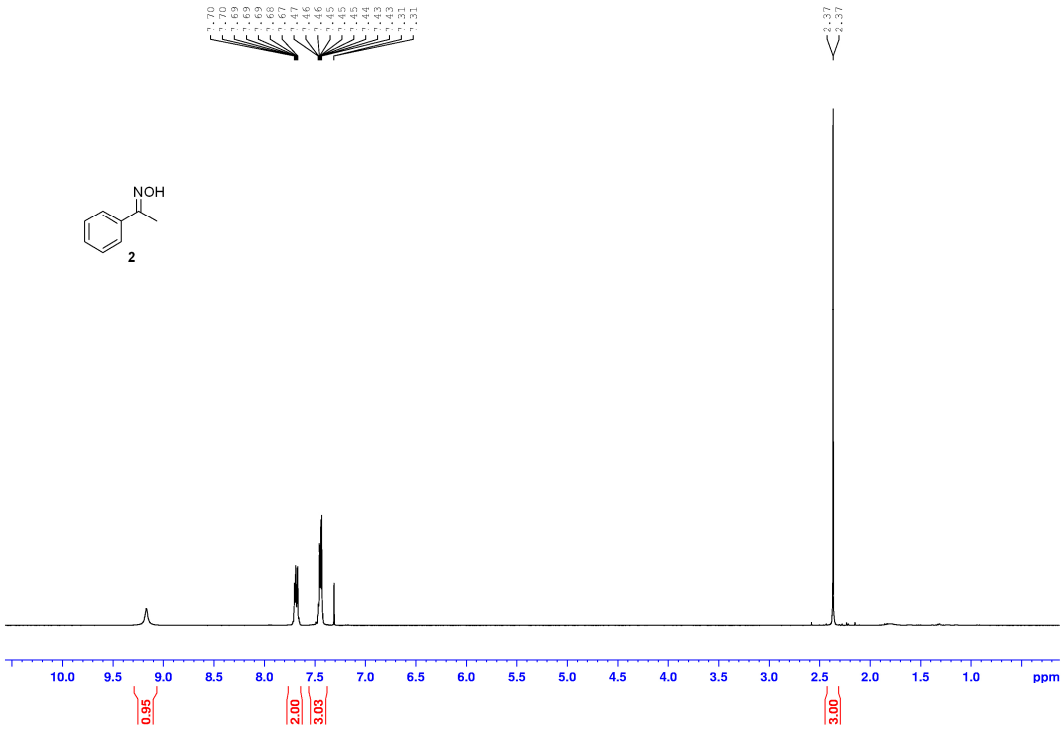
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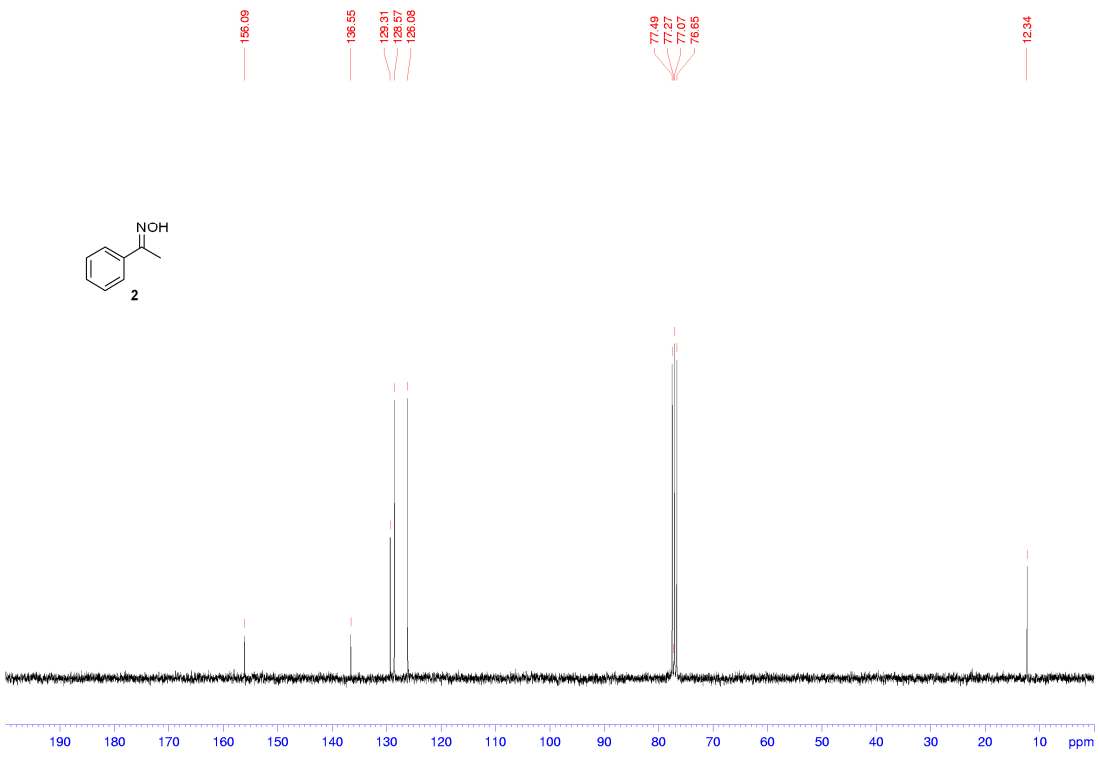
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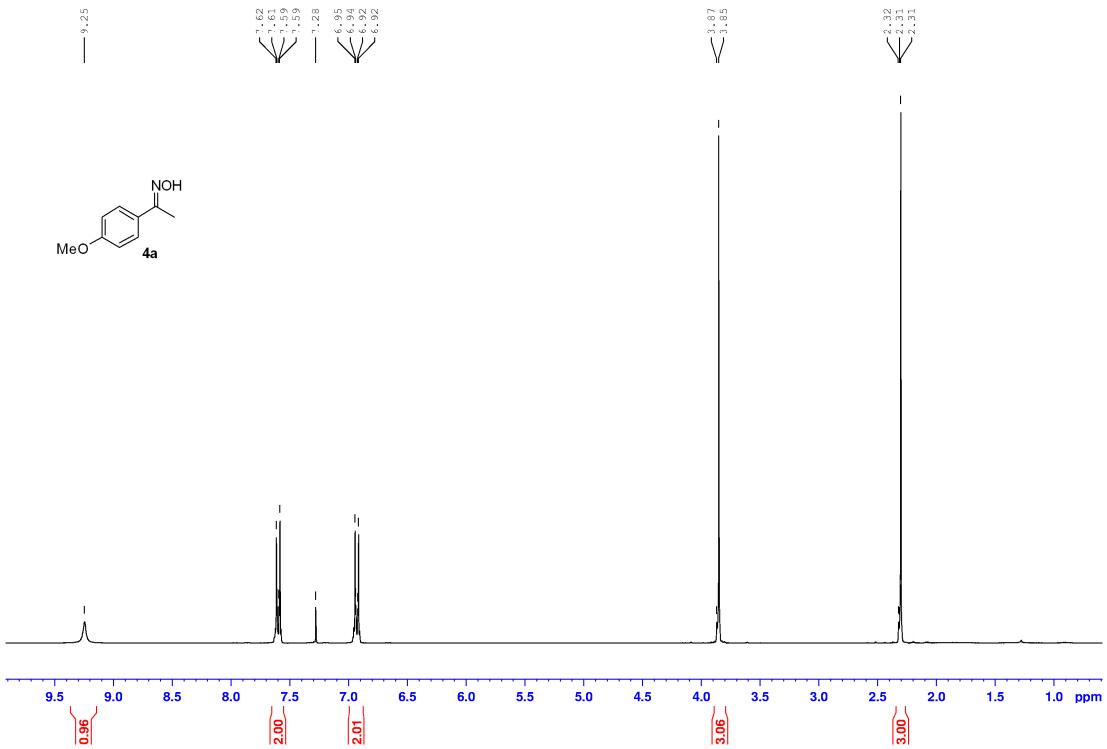
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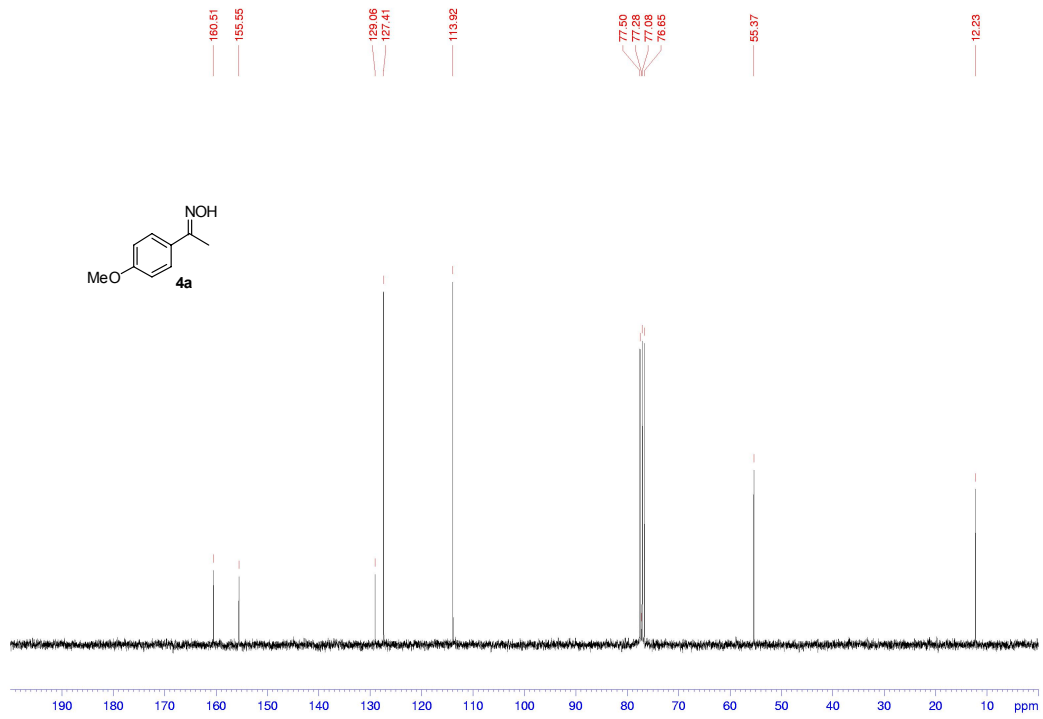
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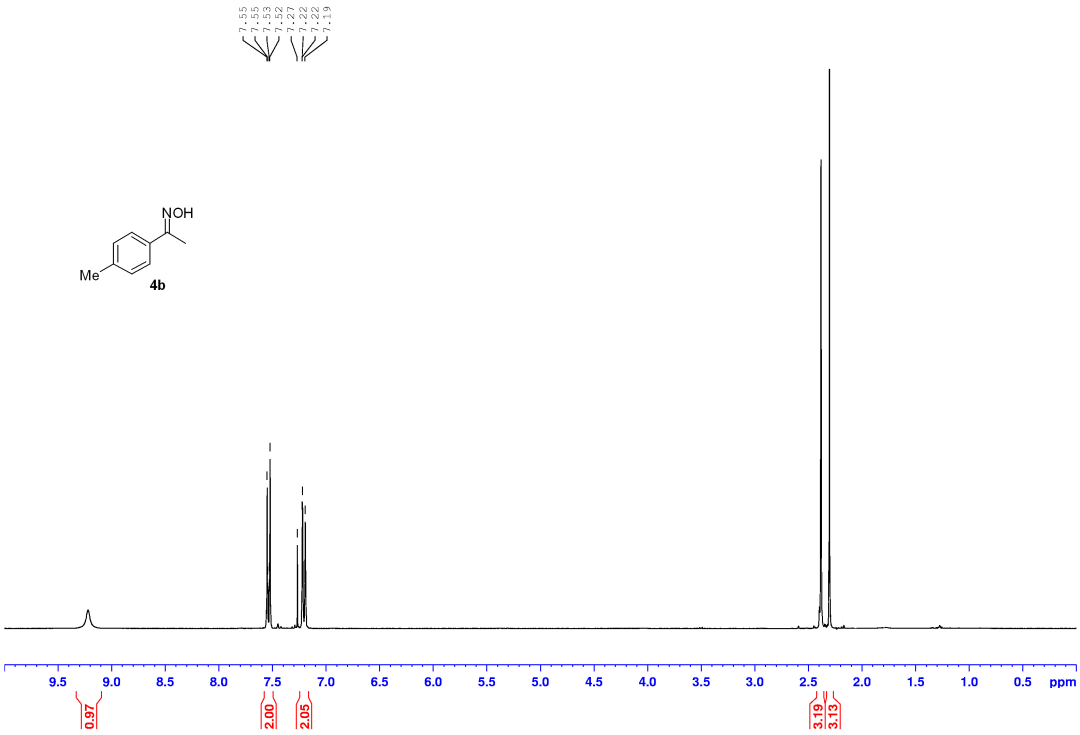
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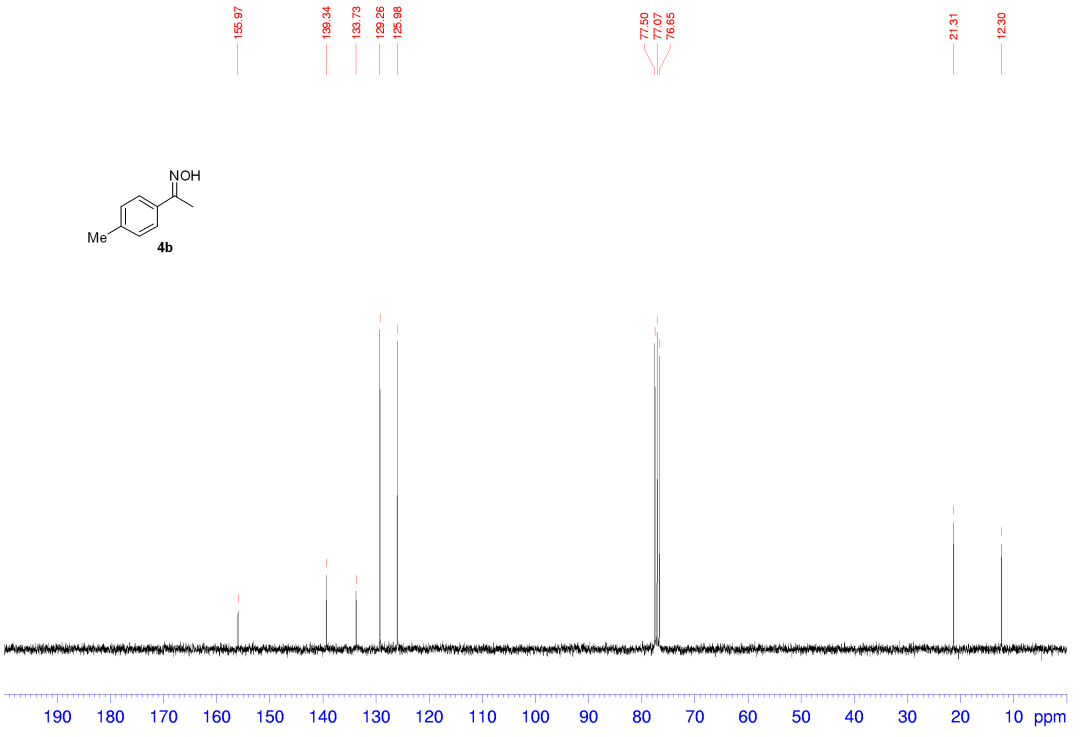
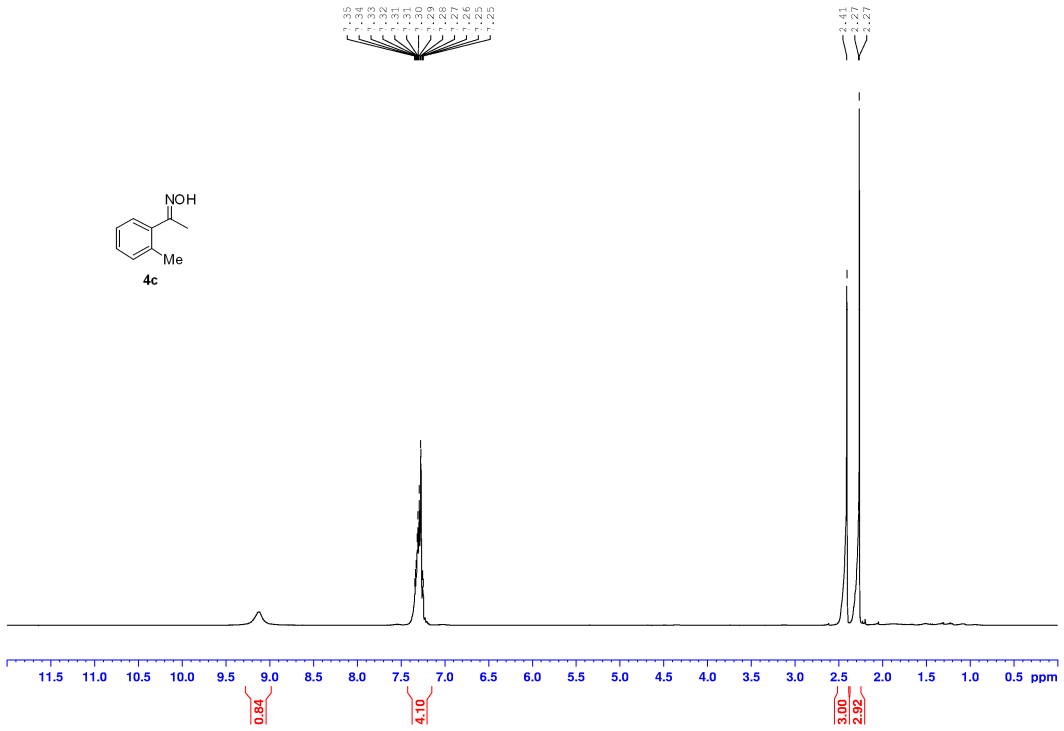
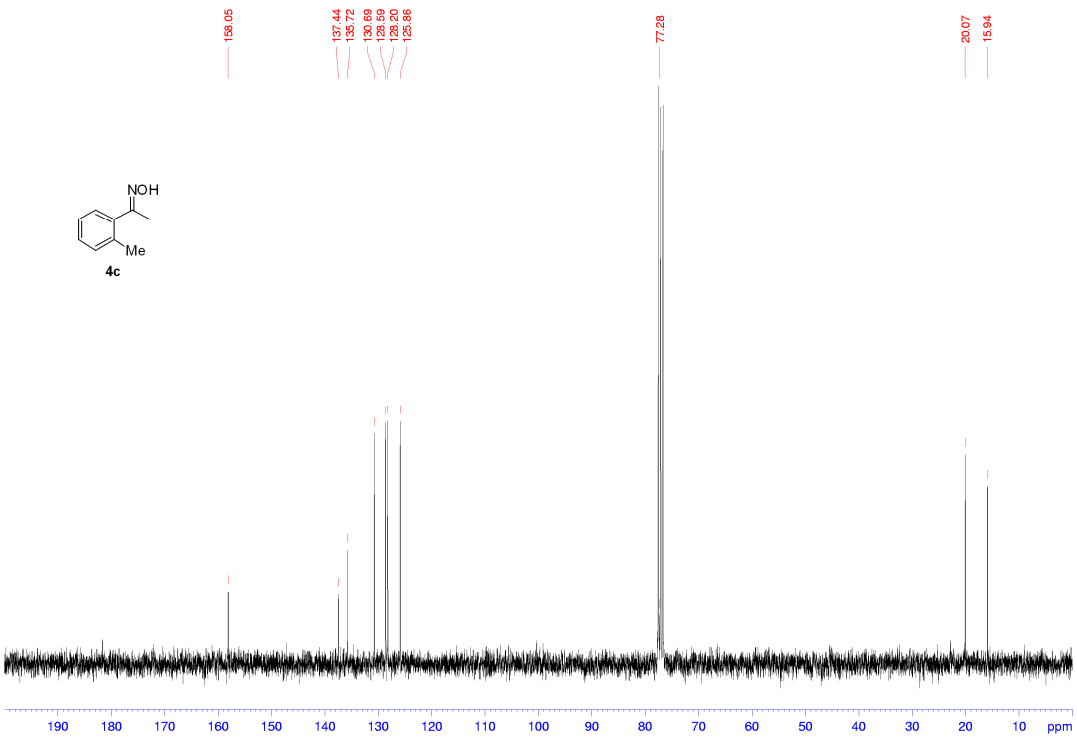
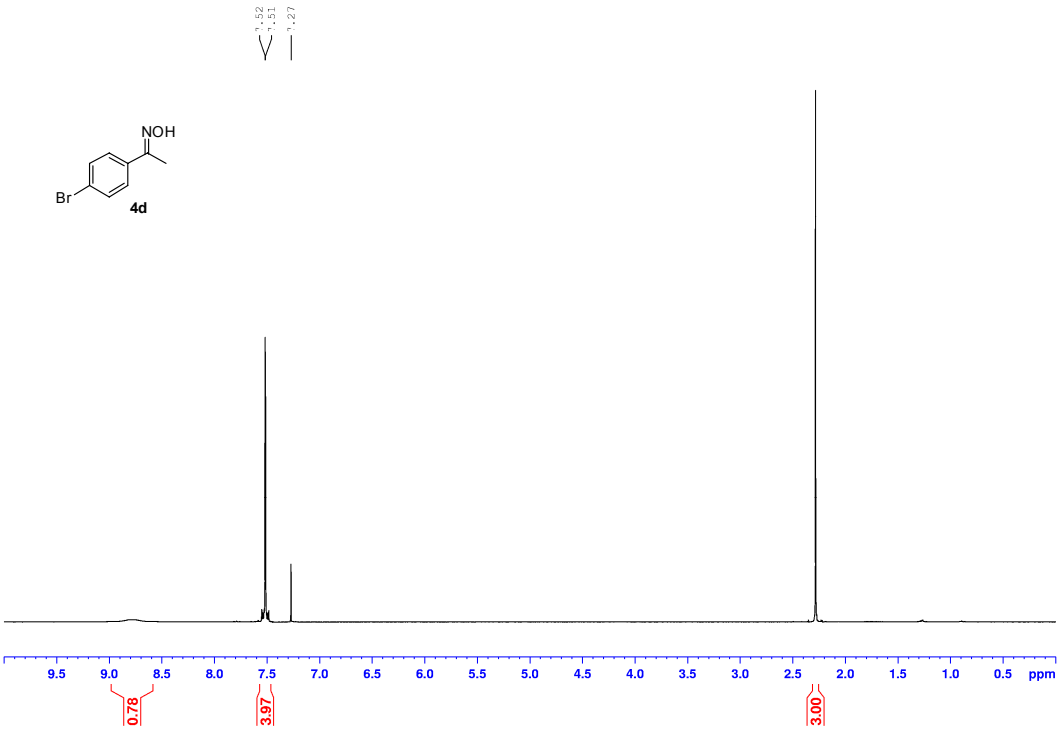
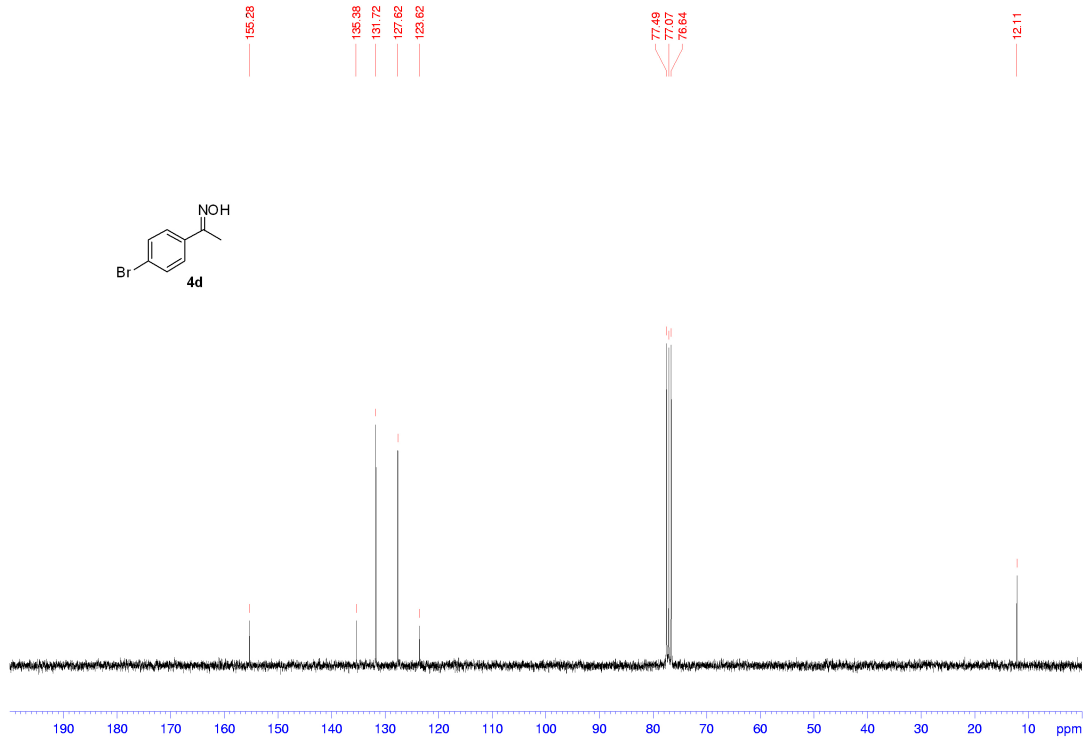
3.13

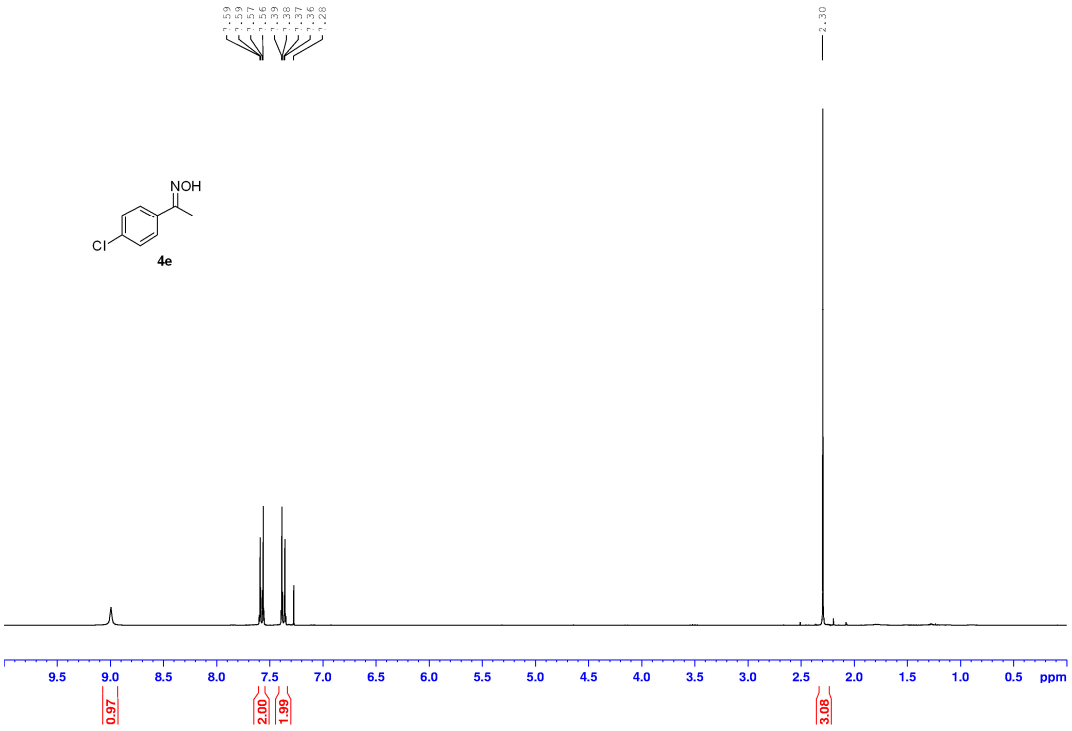

155.97
139.34
133.73
129.26
125.98
77.50
77.07
76.65
21.31
12.30
NOH
Me
4b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

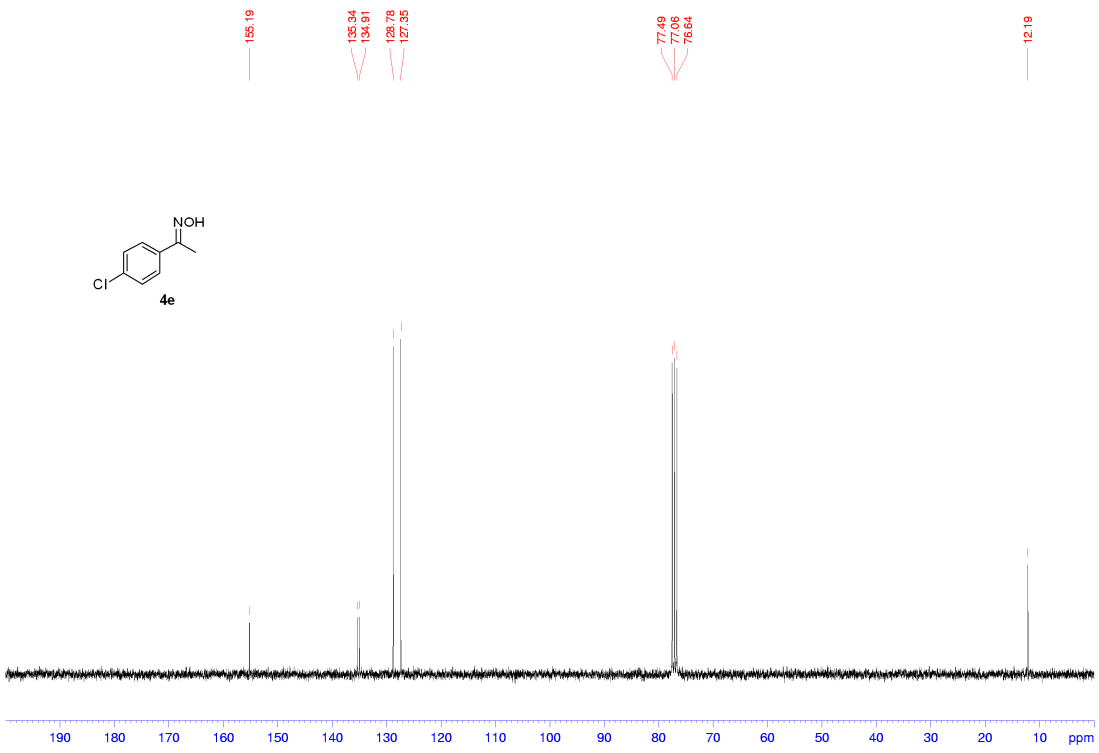

7.35
7.34
7.33
7.32
7.31
7.31
7.30
7.29
7.28
7.27
7.26
7.25
7.25
2.41
2.27
2.27
NOH
Me
4c
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.84
4.10
3.00
2.92

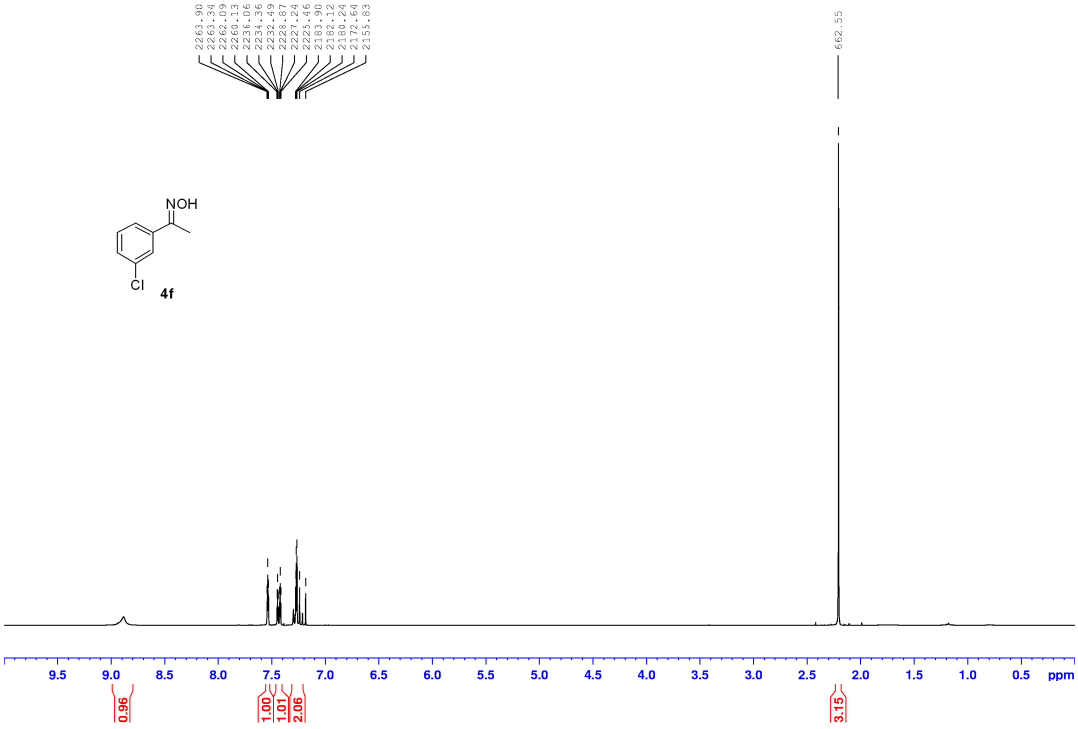

158.05
137.44
135.72
130.69
128.59
128.20
125.86
77.28
20.07
15.94
NOH
Me
4c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

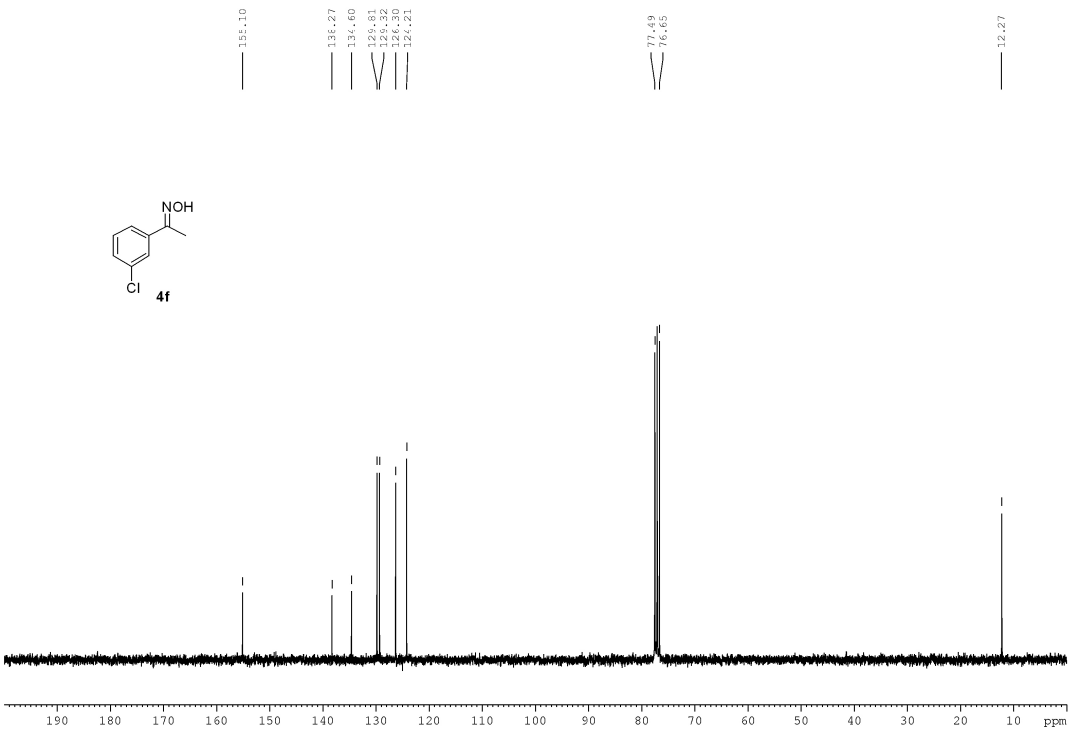

7.52
7.51
7.27
NOH
Br
4d
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.78
3.97
3.00

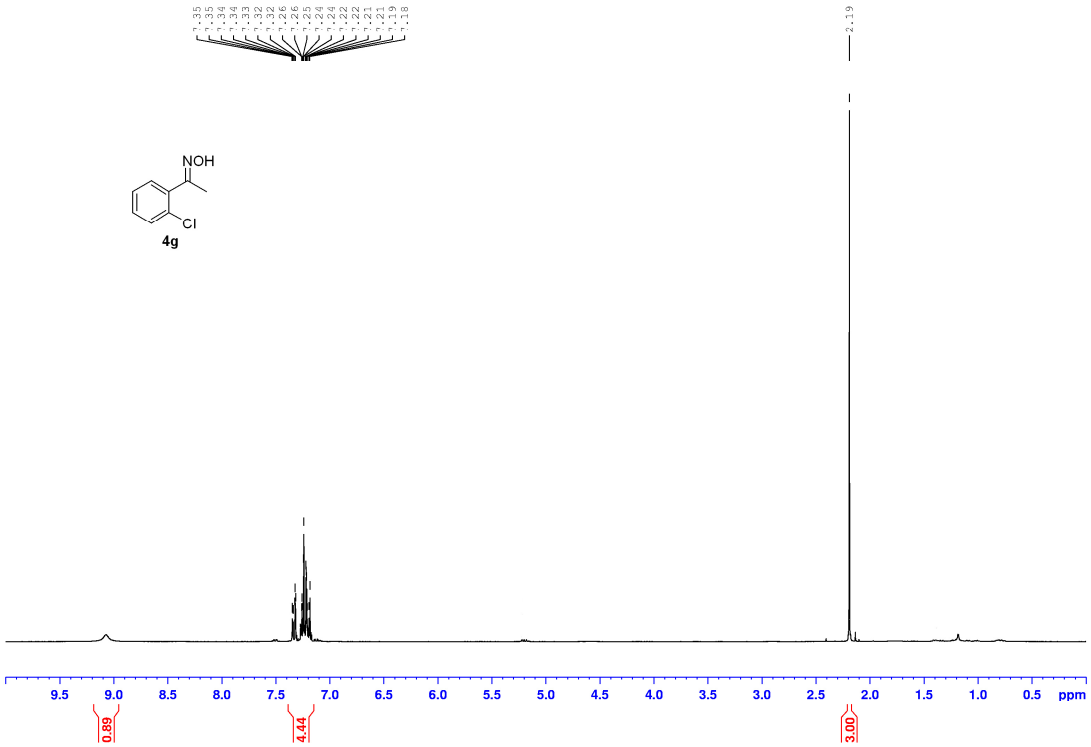

155.28
135.38
131.72
127.62
123.62
77.49
77.07
76.64
12.11
NOH
Br
4d
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

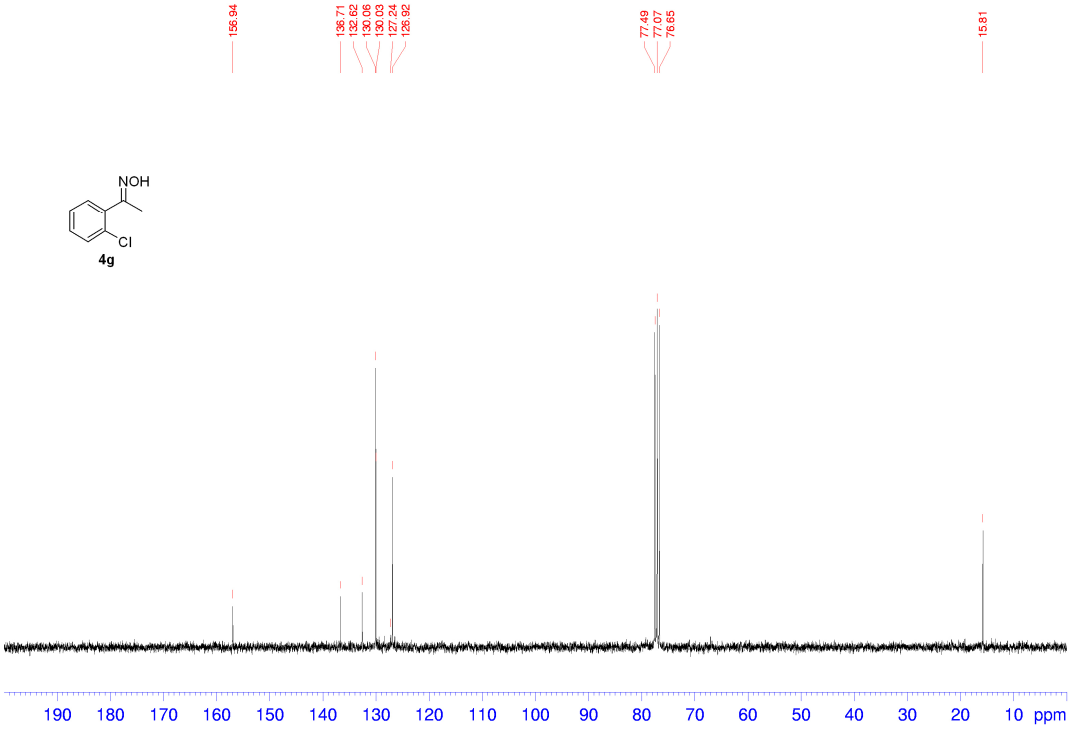

7.59
7.59
7.57
7.56
7.39
7.38
7.37
7.36
7.28
2.30
NOH
Cl
4e
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.97
2.00
1.99
3.08


155.19
135.34
134.91
128.78
127.35
77.49
77.06
76.64
12.19
NOH
Cl
4e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


2263.90
2263.34
2262.09
2260.13
2236.06
2234.36
2232.49
2228.87
2227.24
2225.46
2183.90
2182.12
2180.24
2172.64
2155.83
662.55
NOH
Cl
4f
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.96
1.00
1.01
2.06
3.15


155.10
138.27
134.60
129.81
128.32
126.30
124.21
77.49
76.65
12.27
NOH
Cl
4f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.35
7.35
7.34
7.34
7.33
7.32
7.32
7.26
7.25
7.24
7.24
7.22
7.22
7.21
7.21
7.19
7.18
2.19
NOH
Cl
4g
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.89
4.44
3.00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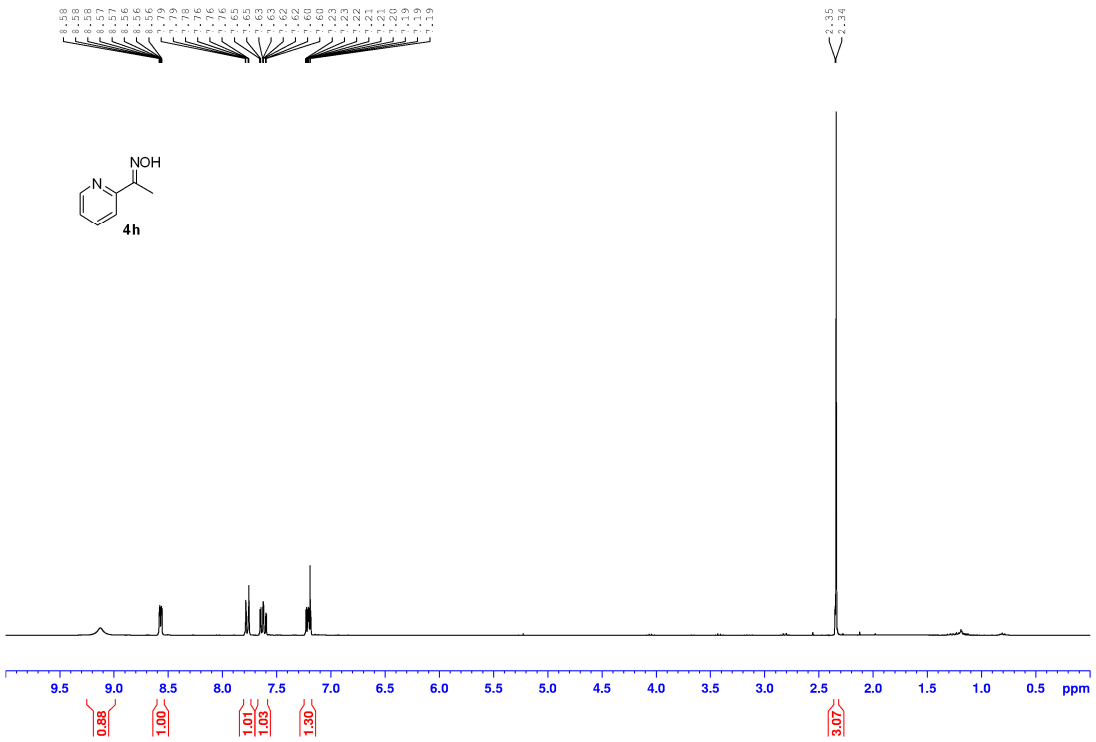
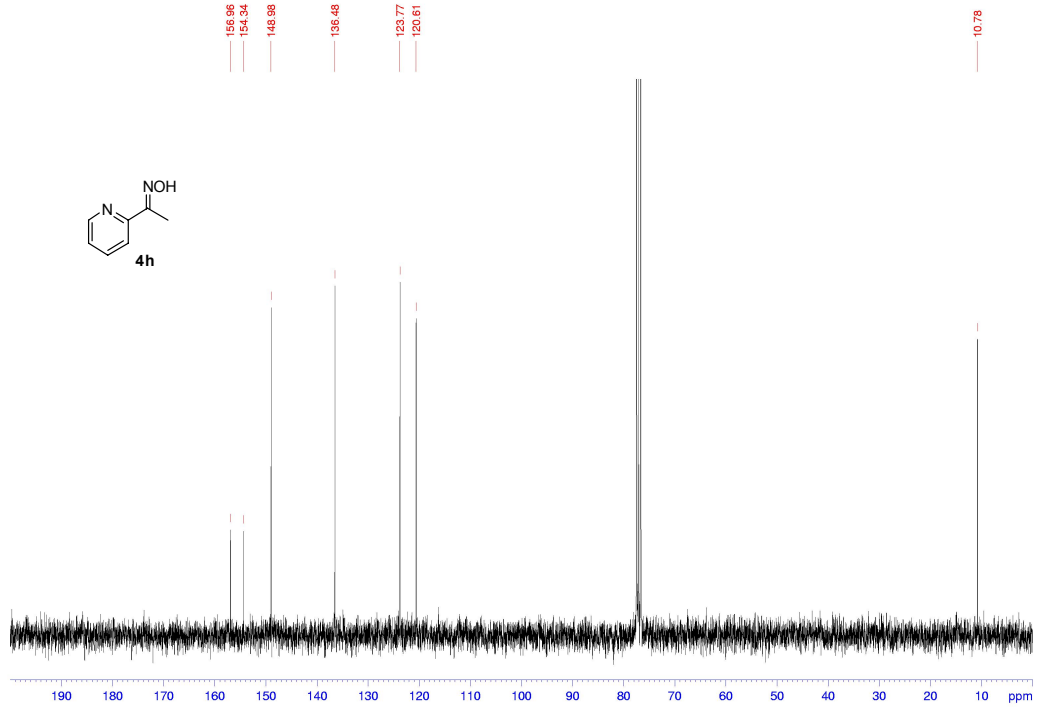
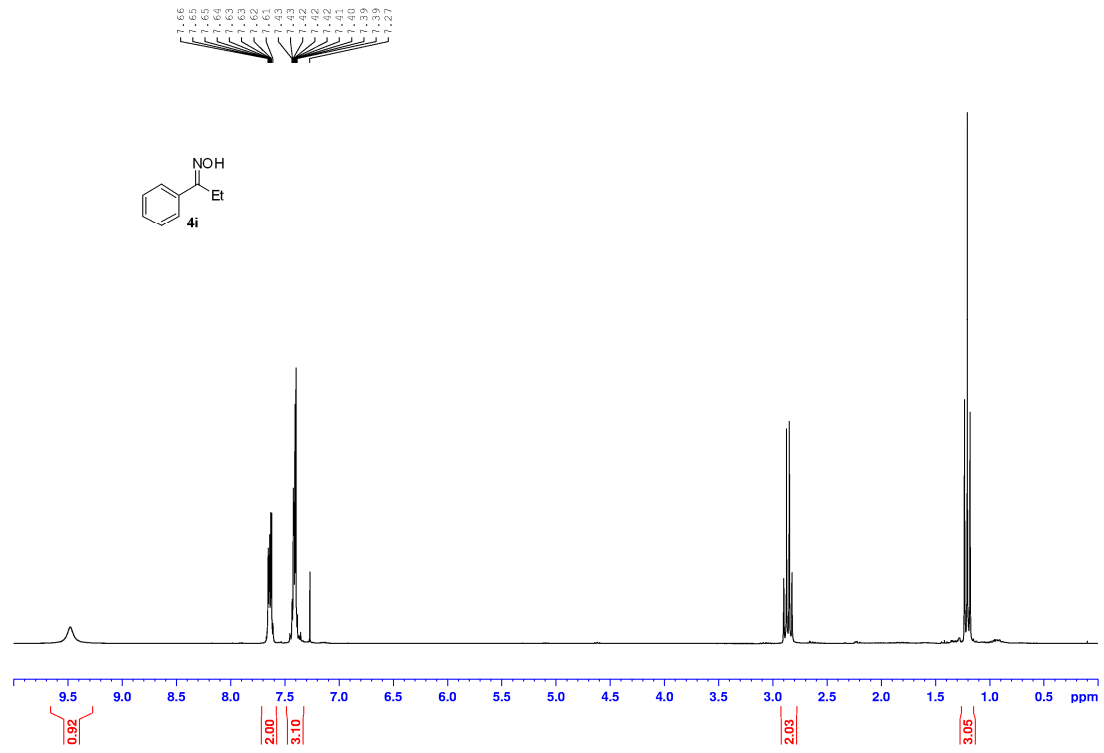
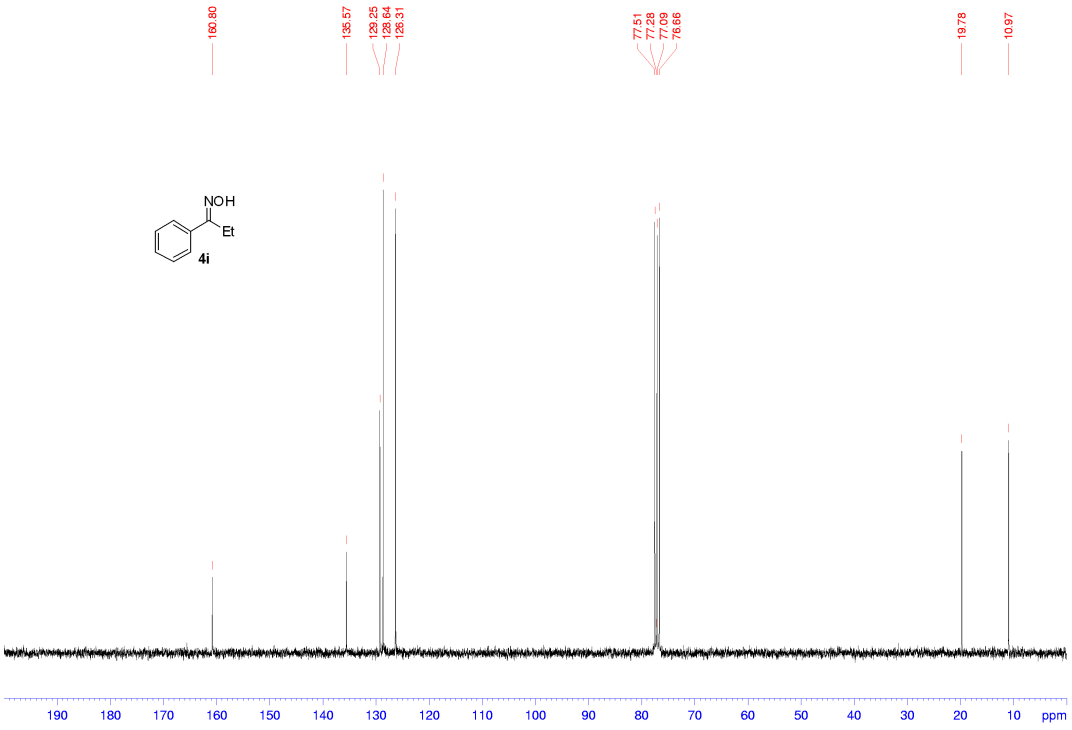
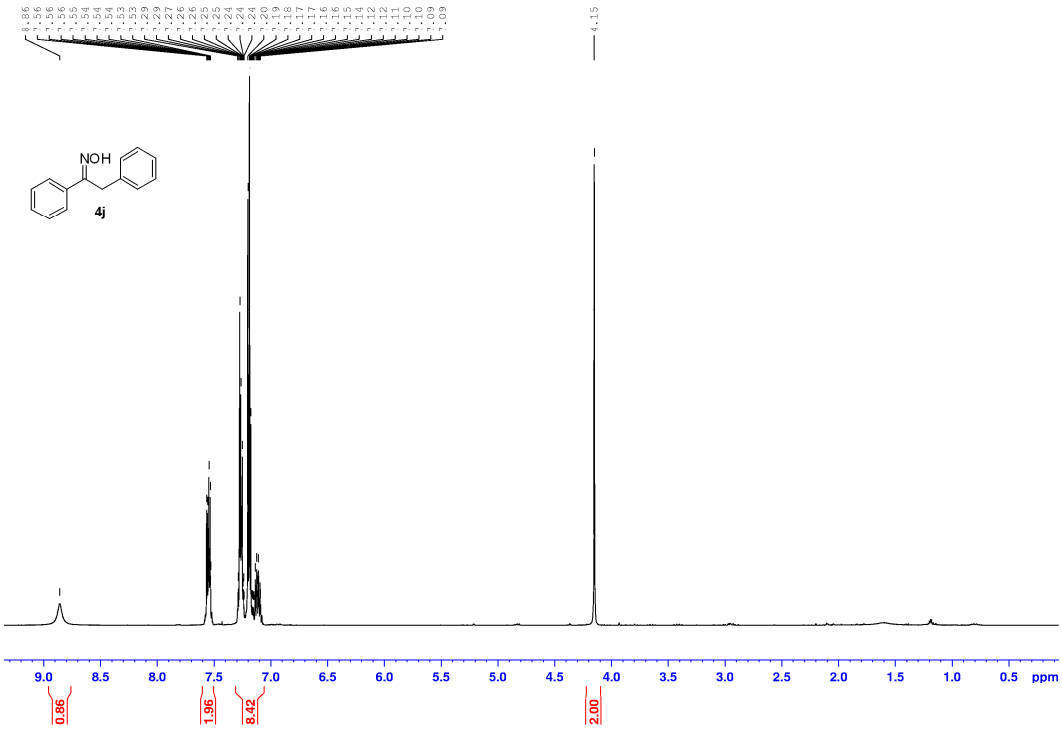
136.71
132.62
130.06
130.03
127.24
125.92
77.49
77.07
76.65
15.81
NOH
Cl
4g
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

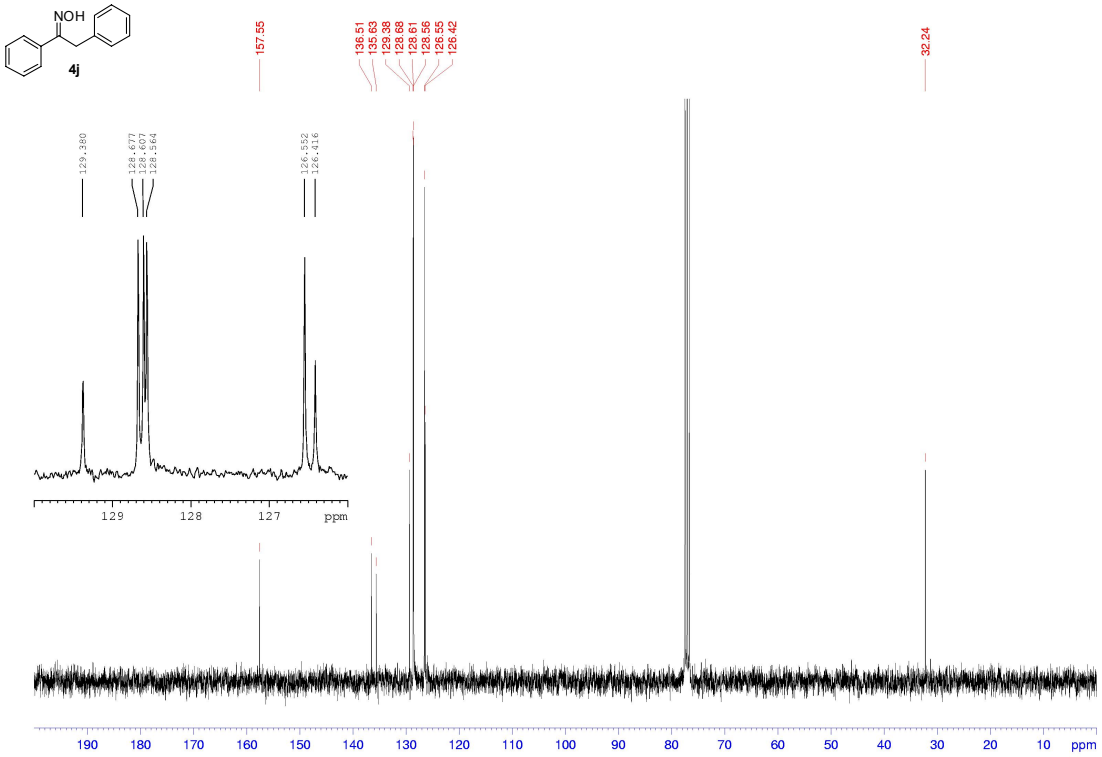

8.58
8.58
8.58
8.57
8.57
8.56
8.56
7.79
7.79
7.78
7.76
7.76
7.76
7.65
7.63
7.63
7.62
7.62
7.60
7.60
7.23
7.22
7.21
7.20
7.19
7.19
7.19
2.35
2.34
NOH
N
4h
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.88
1.00
1.01
1.03
1.30
3.07

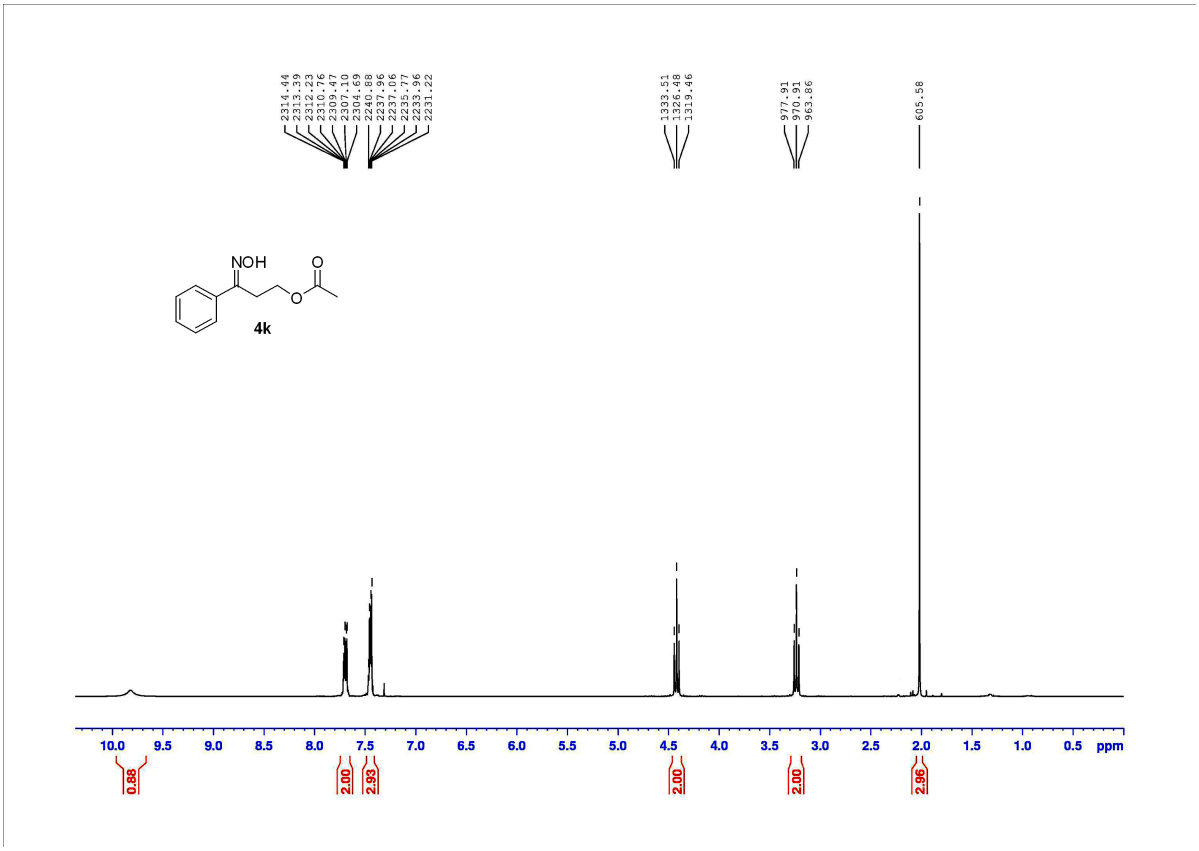

156.96
154.34
148.98
136.48
123.77
120.61
10.78
NOH
N
4h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.66
7.65
7.65
7.64
7.63
7.63
7.62
7.61
7.43
7.43
7.42
7.42
7.42
7.41
7.40
7.39
7.39
7.27
NOH
Et
4i
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.92
2.00
3.10
2.03
3.05


160.80
135.57
129.25
128.64
126.31
77.51
77.28
77.09
76.66
19.78
10.97
NOH
Et
4i
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


NOH
4j
4.15
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.86
1.96
8.42
2.00


NOH
4j
157.55
136.51
135.63
129.38
128.88
128.61
128.56
126.55
126.42
32.24
129.380
128.677
128.607
128.564
126.552
126.416
129
128
127
ppm
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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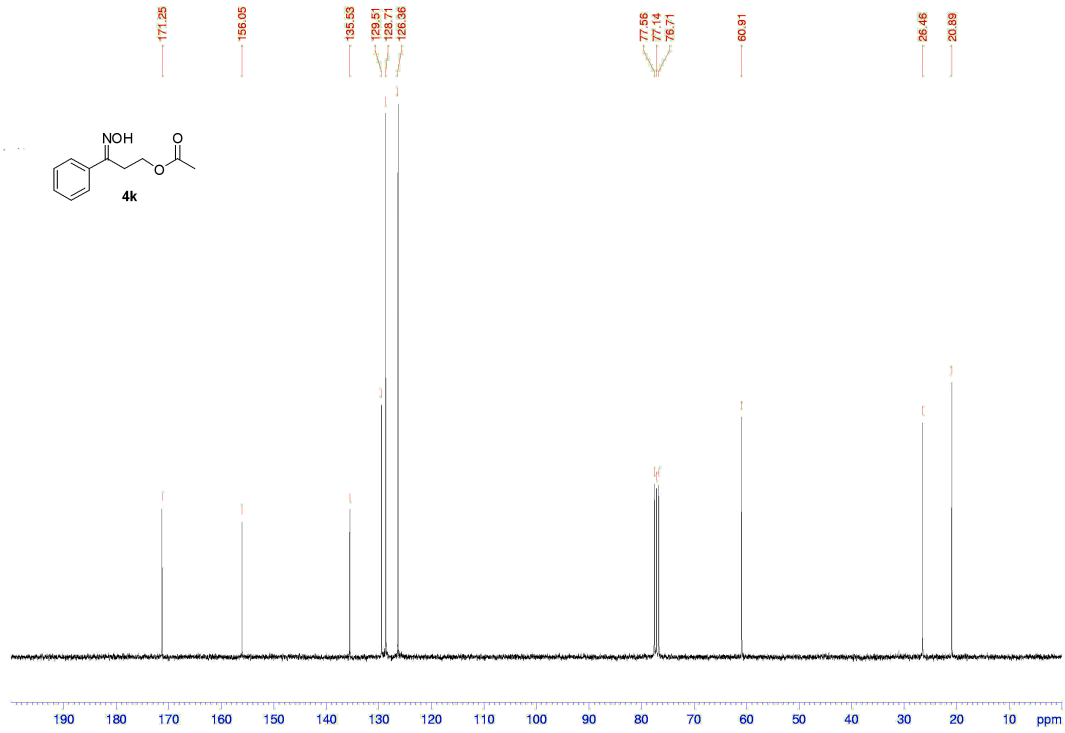
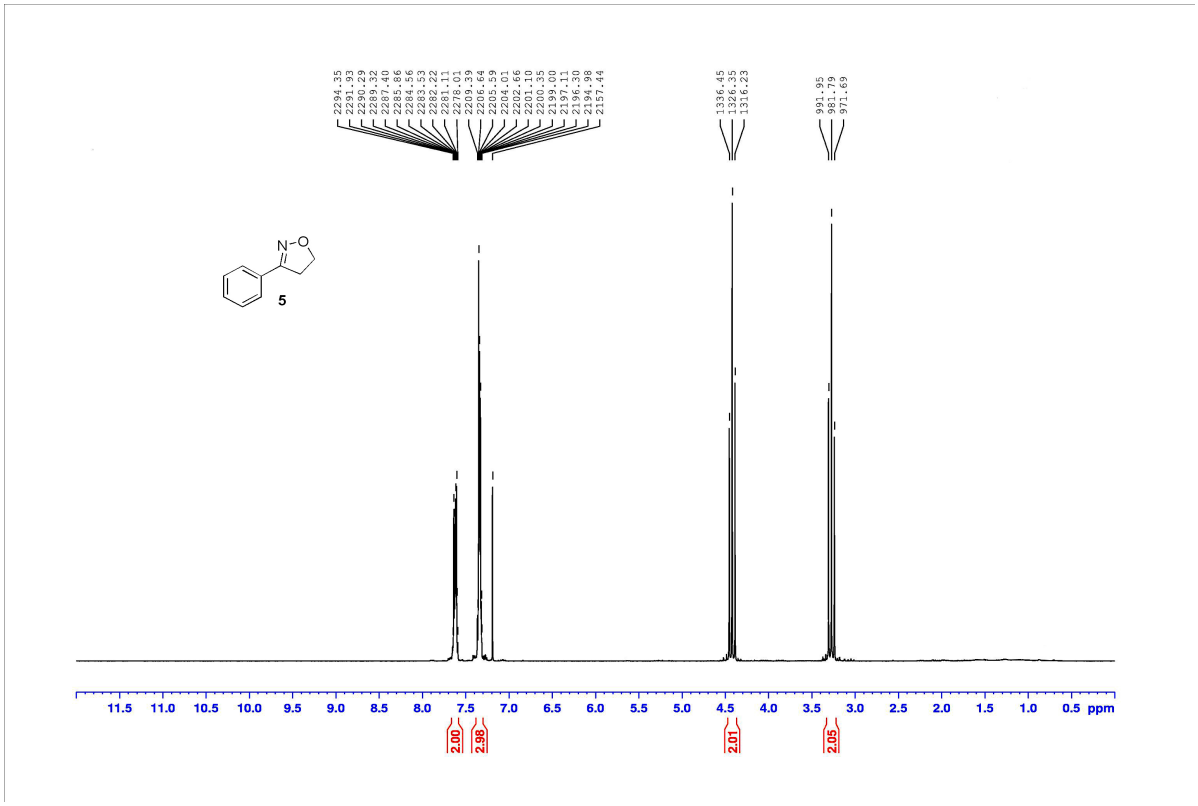
2313.39
2312.23
2310.76
2309.47
2307.10
2304.69
2240.88
2237.96
2237.06
2235.77
2233.96
2231.22
1333.51
1326.48
1319.46
977.91
970.91
963.86
605.58
NOH
O
O
4k
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.88
2.00
2.93
2.00
2.00
2.96


171.25
156.05
135.53
129.51
128.71
126.36
77.56
77.14
76.71
60.91
26.46
20.89
NOH
O
O
4k
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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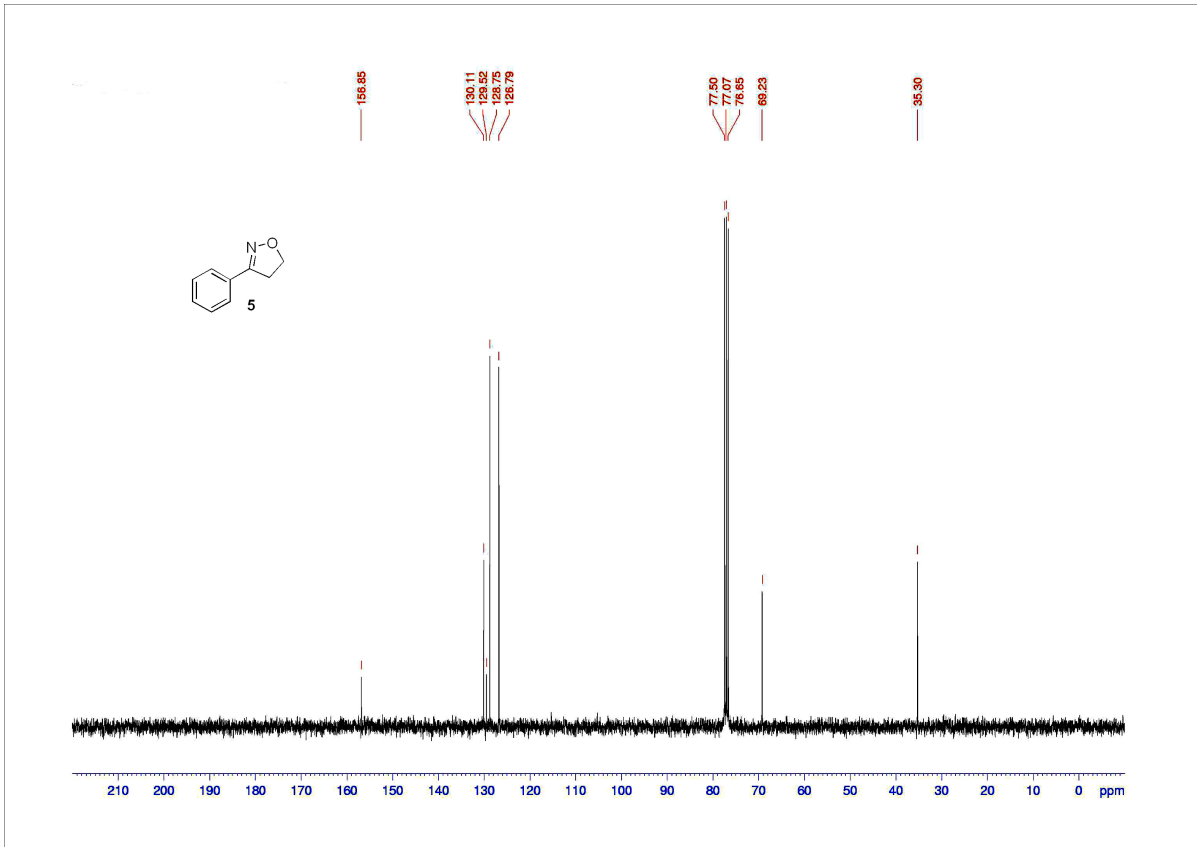
2291.93
2290.29
2289.32
2287.40
2285.86
2284.56
2283.53
2282.22
2281.11
2278.01
2209.39
2206.64
2205.59
2204.01
2202.66
2201.10
2200.35
2199.00
2197.11
2196.30
2194.98
2157.44
1336.45
1336.35
1316.23
991.95
981.79
971.69
N
O
5
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
2.98
2.01
2.05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130.11
129.52
128.75
126.79
77.50
77.07
76.85
69.23
35.30
N
O
5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm